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## Stabilization mechanisms of pure tetragonal zirconia nanocrystallites using an original supercritical-based synthesis route

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Zirconium dioxide (ZrO2) has been of major industrial and scientific interest over the past decades for its wide range of applications, in particular thanks to its catalytic properties. Regardless the multiple synthesis routes employed, undoped bulk ZrO2 is stabilized in the monoclinic structure (m-ZrO2) at ambient temperature and atmospheric pressure. However, these last years, studies showed that it was also possible to stabilize pure t-ZrO2 without cationic substitution. To do so, three main effects are identified in the literature: (i) the presence of oxygen vacancies, (ii) the existence of structural similarities, and (iii) the size effect, with the existence of a critical size below which pure t-ZrO2 is formed.

In this presentation we will introduce the synthesis of quasi pure t-ZrO2 nanocrystals in supercritical fluids using borderline nonhydrolytic sol-gel (B.N.H.S.G.) conditions. The term borderline is used to express the nuance from nonhydrolytic sol-gel (N.H.S.G.) reactions when small amounts of water molecules are generated in situ from solvent (alcohols) decomposition to trigger hydrolysis/condensation. This amount of water being controlled, the reaction kinetics can be tuned and slow down. This enabled us to performed in situ total scattering measurements in order to catch the various stages of the nanocrystal formation upon various conditions (temperature, nature of the alcohols and presence of surfactants). These observations where combined to various ex situ characterizations (Raman, PDF, EXAFS, HR-TEM, XRD, etc.) to discuss the existence of a critical size below which pure t-ZrO2 is formed, as reported in literature and propose a formation mechanism in this specific synthesis route.

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